SANT LONGOWAL INSTITUTE OF ENGINEERING & TECHNOLOGY LABORATORY MANUAL FOR B.E. DEGREE

SUBJECT: APPLIED CHEMISTRY

SUBJECT CODE: BSCH-402



Dr. HEMANT KUMAR ASSISTANT PROFESSOR

DEPARTMENT OF CHEMISTRY SLIET, LONGOWAL (DEEMED UNIVERSITY) Estd. By MHRD, Govt. of India DISTT-SANGRUR, PUNJAB-148106

LABORATORY MANUAL FOR B.E. DEGREE

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BSCH: 402 APPLIED CHEMISTRY LABORATORY

Course Outcomes:

After successful completion of Applied Chemistry-laboratory course, students should be able to:

CO1: Use critical thinking strategies to make connection and association between chemical principles.

CO2: Handle different type of instruments for chemical analysis.

CO3: Develop skills in building technical competence through experimental methods.

CO4: Have practical knowledge of instrumental method of analysis.

CO5: To develop basic knowledge on precision, accuracy and performance in analysis.

| BSCH- | PO | PS | PS |
|-------|----|----|----|----|----|----|----|----|----|----|----|----|----|----|
| 402 | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 01 | 02 |
| CO1 | 3 | 3 | 2 | 3 | 2 | 3 | 3 | N | 3 | 2 | 3 | 3 | S | N |
| CO2 | 3 | 3 | 3 | 3 | 3 | 2 | 2 | N | 3 | N | 3 | 3 | S | Ν |
| CO3 | 3 | 3 | 2 | 3 | 3 | 2 | 2 | N | 2 | N | 2 | 3 | S | Ν |
| CO4 | 3 | 2 | 3 | 3 | 3 | 3 | 2 | N | 2 | 2 | 3 | 3 | S | N |
| C05 | 2 | 3 | 2 | 2 | 2 | 1 | 2 | N | 2 | 1 | 1 | 3 | S | N |

List of Experiments: (Any twelve to be performed)

1. Determination of Total Hardness of water (tap, lake, pond, river) using standard EDTA solution and Eriochrome Black T (EBT) indicator.

- 2. Determination of Available Chlorine in treated and untreated water titrimetrically.
- 3. Determination of Available Chlorine in Bleaching Powder titrimetrically.
- 4. Preparation and characterization of nylon 66.
- 5. Preparation and characterization of polystyrene.
- 6. Preparation of urea-formaldehyde resin.

- 7. Preparation of phenol-formaldehyde resin (Bakelite).
- **8.** To determine the molecular weight of a given polymer (polystyrene) by using viscometric method.

- 9. To find the eutectic point for two component system by cooling curve method.
- **10.** To determine the distribution coefficient of iodine between CCl₄ and water at room temperature.
- 11. Identification of functional groups by FT-IR spectroscopy.
- 12. To determine $\Lambda_{(max)}$ (wave length of maximum absorption) of solution of KMnO4 using a spectrophotometer.
- 13. Determination of concentration of an unknown sample by UV spectroscopy.
- **14.** Determination of coefficient of viscosity of the given liquids by Ostwald's viscometer method.
- **15.** Investigation of rusting of iron in different conditions.
- 16. Investigation of the effect of metal coupling on rusting of iron.

EXPERIMENT NO. : 01

AIM: Determination of total hardness of water (tap, lake, pond, river) using standard EDTA solution and Eriochrome Black T (EBT) indicator.

APPARATUS: Conical flask, burette, pipette, and measuring cylinder.

CHEMICALS: 0.2N EDTA, ammonium buffer solution of $NH_4Cl + NH_4OH$ (pH 9-10), standard hard water (0.02 N, 1 gram CaCO₃ per litre), Eriochrome Black T indicator (EBT) and water sample (tap water, unknown hardness).

PRINCIPLE:

The property of water which restricts or checks the lather formation with soap is called hardness. In other words, the presence of multivalent cations, mostly calcium and magnesium ions, in water is referred to as hardness of water. Hardness is of two types: Temporary or carbonate hardness which can be removed by boiling and Permanent or Non carbonate hardness which cannot be removed by boiling. The hardness is usually expressed in parts of CaCO₃ equivalent or calcium and magnesium salts per million parts of water i.e. in ppm.

The hardness of water can be determined by complexometric titration using Ethylene diammine tetra acetic acid (EDTA). EDTA in the form of its di-sodium salt forms complex with Ca^{2+} and Mg^{2+} ions of water sample. When Eriochrome Black T (EBT) indicator is added to the hard water at pH around 9–10, it gives wine red colored unstable complex with Ca^{2+} and Mg^{2+} ions of water sample. When this wine red colored complex is titrated against EDTA solution of known strength the Ca^{2+} and Mg^{2+} ions forms stable metal complex with EDTA and color changes from wine red to blue (color of EBT indicator) at the endpoint.



So titration at pH about 9–10 using EBT indicator gives the total amount of Ca^{2+} and Mg^{2+} ions in the water sample. Titration against EDTA at pH around 12.5 gives the hardness due to Ca^{2+} only. A pH of about 12.5 required for this titration can be obtained by

adding diethyl amine base with 3- 4 drops of calcon indicator or NaOH base with murexide indicator. At this high pH, the Mg^{2+} ion is quantitatively precipitated as $Mg(OH)_2$ and Ca^{2+} ion alone can be estimated by complexometric method using EDTA. At the end point color changes from pink to pure blue.

Structure of di-sodium salt of EDTA:



Structure of M-EDTA complex



Structure of EBT Indicator:



PROCEDURE:

Standardization of EDTA solution with Standard Hard Water:

1. Take 20 ml of standard hard water marked as V_1 ml in a conical flask. Add 5 ml of buffer solution and 2 to 3 drops of EBT indicator, the color of solution turns wine red.

- 2. Titrate the solution in the flask solution against standard EDTA solution from the burette until the color changes from wine red to blue at the end point.
- 3. Take at least three concordant readings. Let the volume of EDTA solution used = V_2 ml
- 4. Titrate similarly the unknown hard water sample and repeat the same procedure to find out volume of EDTA solution used. Let the volume of EDTA used with unknown hard water = V_3 ml

OBSERVATION TABLE:

For Standard Hard Water (SHW):

Normality of standard hard water used as $N_1 = 0.02 \text{ N}$

Standard hard water volume used for each titration as $V_1 = 20$ ml.

0.2 N EDTA solution volume used for each titration = V_2 ml

| S. No. | Volume of standard hard water (SHW) (ml) | Volume of EDTA solution (ml) | Concordant reading (ml) |
|--------|---|---------------------------------|----------------------------|
| 1 | 20 ml | | |
| 2 | 20 ml | | V ₂ |
| 3 | 20 ml | | |

For Unknown Hard Water sample (UHW):

| S. No. | Volume of unknown hard water (UHW) (ml) | Volume of EDTA solution (ml) | Concordant reading (ml) |
|--------|--|---------------------------------|----------------------------|
| 1 | 20 ml | | |
| 2 | 20 ml | | V ₃ |
| 3 | 20 ml | | |

CALCULATIONS

For standard hard water:

SHW= EDTA

 $N_1V_1 = N_2V_2$

 $N_2 = N_1 V_1 / V_2$

For unknown hard water sample:

Normality of unknown hard water

EDTA = UHW

 $N_2V_2 = N_3V_3$

 $N_3 = N_2 V_2 / V_3$

Hardness of unknown hard water = weight of salt causing hardness x equivalent weight of $CaCO_3$ in terms of $CaCO_3$ equivalents equivalent weight of salt

= Normality of unknown hard water x equivalent weight of CaCO₃

=.....gm/litre

= x 1000 mg/ litre

RESULT: The total hardness of given unknown water sample (tap water) is

mg/litre.

PRECAUTIONS

- 1) Washed all the glass apparatus thoroughly with distilled water before use.
- 2) Rinsed the burette and pipette properly with solution to be taken in it.
- 3) The solutions required for experiment should be freshly prepared.
- 4) Shaking of conical flask should be proper during titration.
- 5) The titration flask should be watched properly to identify the color change at the endpoint.

EXPERIMENT NO. : 02

AIM: Determination of Available Chlorine in treated and untreated water titrimetrically.

APPARATUS: Burette & burette stand, porcelain Tile, pipettes with elongated tips, pipette bulb, wash bottle, 250 mL graduated cylinder, 500 mL conical flask (Erlenmeyer flask)

CHEMICALS: Acetic acid (glacial), Potassium iodide, KI crystals, Sodium thiosulphate, Starch indicator, Distilled water

PRINCIPLE: Chlorination is primarily adopted to destroy or deactivate disease-producing microorganisms in the public water supplies and polluted rivers. Chlorine is usually added to water in gaseous form or as sodium or calcium hypochlorite. When chlorine is added to water, some of the chlorine reacts first with organic materials and metals in the water and is not available for disinfection (this is called the chlorine demand of the water). The remaining chlorine concentration after the chlorine demand is accounted for is called total chlorine. Total chlorine is further divided into: 1) the amount of chlorine that has reacted with nitrates and is unavailable for disinfection which is called combined chlorine and, 2) the free chlorine, which is the chlorine available to inactivate disease- causing organisms, and thus a measure to determine the potability of water

The starch-iodide titration method, one of the oldest methods for determining chlorine, is very non-specific for oxidants and generally is used for total chlorine testing at levels above 1 mg/L Cl₂. Chlorine will liberate free iodine from potassium iodide (KI) solutions at pH 8 or less. The liberated iodine is titrated with a standard solution of sodium thiosulphate $(Na_2S_2O_3)$ with starch as the indicator.

 $Cl_2 + KI \rightarrow 2KCl + I_2$

$$I_2 + Na_2S_2O_3 \rightarrow Na_2S_4O_6 + 2NaI$$

 I_2 + Starch solution \rightarrow Blue color

This method is based on reaction with thiosulfate solution. The end point of the titration is indicated by the disappearance of the blue- colored, starch-iodide complex.

PROCEDURE

They are two different methods available to estimate the residual chlorine.

- Iodometric Method: This method is more precise than colorimetric method where residual concentration exceeds 1mg/L, but for lower concentration it is not so accurate.
- 2) Stabilized Neutral Ortho-Toluidine method: This method is useful to determine free available chlorine and combine chlorine. This method is sensitive to low residual chlorine concentrations.

PREPARATION OF REAGENTS

Sodium Thiosulphate solution (0.01N)

Weigh approximately 2.482 g of sodium thiosulphate (N/100)

Transfer to the beaker and dissolve it in boiled distilled water.

Transfer it to the standard flask and make it up to 1000 mL.

- 1. Rinse the burette with sodium thiosulphate and then fill the burette with sodium thiosulphate.
- 2. Fix the burette to the stand.
- 3. Take 20mL of a given water sample in a conical flask.
- 4. Add 2 ml acetic acid. To acidify the sample. It is used to reduce the pH between3 and 4 in the conical flask.
- 5. Add about 1g (10ml) Potassium iodide (KI) measured using the spatula and dissolve it by thoroughly mixing it with stirring rod.

6. Perform the titration quickly, since iodine liberate faster.

- Titrate the solution with standard Na₂S₂O₃ solution until the yellow color of liberated Iodine is almost faded out. (Pale yellow color)
- Add 1 ml of starch solution and continue the titration until the blue color disappears.
- 9. In many cases residual chlorine is very low and starch needs to be added before starting up the titration.

10. Note down the burette reading (to know the volume of sodium thiosulphate added).

Burette Solution: Sodium thiosulphate

Pipette Solution: Sample

Indicator: Starch

End point: Disappearance of blue color

OBSERVATION TABLE

| S. No. | Volume of | Burette Re | Volume of | |
|--------|-------------|-----------------|---------------|-------------------|
| | sample (mL) | | | Na2S2O3 used (ml) |
| | | Initial reading | Final reading | |
| | | (ml) | (ml) | |
| 1. | | | | |
| 2. | | | | |
| 3. | | | | |

CALCULATION

Water Sample = Hypo $(Na_2S_2O_3)$

 $N_1V_1=N_2V_2$

 $N_1 = N_2 V_2 / V_1$

Residual Chlorine = $N_1 X$ equivalent weight of chlorine

=x 35.5

=gm/L

=x 1000 mg/L

RESULT

The amount of available chlorine in the given sample of water ismg/liter

PRECAUTIONS:

- Do not expose the potassium iodide crystals in the air. If possible do the experiment in iodine flask instead of conical flask.
- Chlorine in water solutions is not stable. As a result, its concentration in samples decreases rapidly.
- 3. Samples to be analyzed for chlorine cannot be stored or preserved. Tests must be started immediately after sampling. Therefore, samples taken for the chlorine residual test must be grab samples only and excessive agitation must be avoided.
- 4. Exposure to sunlight or other strong light, air, or agitation will further reduce the quantity of chlorine present in solutions.

AIM: Determination of available chlorine in bleaching powder titrimetrically.

APPARATUS: Burette, Pipette, Conical Flask, Funnel, Beaker, Dropper, Filter paper.

CHEMICALS: Standard sodium thiosulphate, potassium iodide 10%, glacial acetic acid, bleaching powder and starch solution.

INDICATOR: Freshly prepared starch solution.

END POINT: Deep blue color disappears.

PRINCIPLE:

Available chlorine is the amount of chlorine liberated by the action of an acid on bleaching powder (CaOCl₂) as shown in the following reaction.

 $CaOCl_2 + 2CH_3COOH \rightarrow (CH_3COO)_2Ca + H_2O + Cl_2 \uparrow$

The liberated chlorine oxidize KI and liberated out the Iodine in equal amount that may be titrated against Standard hypo solution using freshly prepared starch solution as an indicator.

 $Cl_2 + 2KI \rightarrow 2KCl + I_2$

 $I_2 + 2 \ Na_2S_2O_3 \rightarrow 2NaI + Na_2S_4O_6$

PROCEDURE:

Step I: PREPARATION OF BLEACHIMG POWDER SOLUTION

1. Weigh out accurately 01 gm of bleaching powder (01gm in 01L) in a cleaned and pre-weighed weighing bottle.

2. Transfer it into a mortar, crush with a pestle and little distilled water.

- 3. Make a thin paste of bleaching powder sample with distilled water.
- 4. Transfer the paste into 250 ml volumetric flask.
- 5. Wash the mortar and pestle with distilled water and transfer it into the flask to make 250ml.
- 6. Shake well to get homogenous suspension of bleaching powder.

Step II: ESTIMATION OF AVAILABLE CHLORINE

 Wash the burette with distilled water and rinse with standard hypo solution then fill the burette.

- 2. Pipette out 20ml of homogenous solution of bleaching powder in the conical flask.
- 3. Put 10ml of potassium iodide (KI) and about 02ml test tube of glacial acetic acid into the flask.
- 4. Titrate the liberated iodine against standard hypo solution till a pale yellow color is obtained.
- 5. Now added 2-3 drops of starch indicator to get a deep blue color.
- 6. Continue adding the hypo solution till blue color disappears.(End point)
- 7. Repeat the experiment to get the concordant reading.

OBSERVATION TABLE:

| S. | Volume of | Burette R | eading (ml) | Volume of | Concordant Deading |
|------|----------------------------|--------------------|------------------|-----------|-----------------------|
| INO. | powder V ₁ (ml) | Initial Reading | Final Reading | (ml) | V ₂ (ml) |
| 1 | | | | | |
| 2 | | | | | |
| 3 | | | | | |

CALCULATION:

Bleaching Powder Vs Sodium thiosulphate solution

 $N_1 \, V_1 = \, N_2 \, V_2$

 $N_1 \times 10 = 1/10 \times V_2$

 $N_1 = V_2/100$

Strength of available chlorine = $N_1 \times 35.5$ (Eq Wt of chlorine)

% of Avaiable chlorine = $N_1 \ge 35.5 \ge \frac{250}{1000} \ge \frac{100}{W}$

RESULT:

Amount of available chlorine present in bleaching powder is%.

PRECAUTIONS:

- 1. Wash the glass apparatus with the distilled water.
- 2. All the reagents should be freshly prepared.
- 3. Pipette out 10ml of bleaching powder solution accurately.
- 4. The amount of starch indicator should be same in all the titration.
- 5. The end point of the titration should be carefully observed.

EXPERIMENT NO.: 04

AIM: Preparation and characterization of nylon 66.

APPARATUS: Dropper, measuring cylinder, beakers, test tubes, glass rod, water bath, etc.CHEMICALS: Hexamethlenediamine (1,6-hexanediamine), Adipoyl chloride, SodiumHydroxide pellets. Solvent: tap water and cyclohexane.

THEORY: nylon 6,6 is an aliphatic polyamide that is synthesized using A-A/B-B stepgrowth condensation-polymerization. The two monomers involved in this polymerization are hexamethylenediamine and adipoyl acid , each of which is bifuctional (i.e., two ends of each are reactive), and each end contains the same functionality (i.e., A or B functional groups).

The first step in the reaction between a molecule of hexamethylenediamine and a molecule of adipoyl chloride . The hydrogen atom of the amine group belonging to hexamethylenediamine forms a hydrochloric acid (HCl) molecule with the chloride from the acid functional group. The remaining adipoyl chloride molecule and the hexamethylenediamine molecule will join together to form a larger molecule (polymer). The molecule formed has an acid group at one end and an amine group at the other and is a nylon 6,6 unit.



Nylon 66

PROCEDURE:

Prepare 25 mL of a 0.35 M aqueous solution of hexamethylenediamine (1,6-hexanediamine). For example, you would need 1.0 gram of 1,6-hexanediamine in 25 mL of water to make a 0.35 M solution. To do this, weigh out the solid 1,6-hexanediamine on paper using a balance.

- Add water. Use a glass stirring rod to mix the solid until it is completely dissolved in water. Measure 10 mL of this solution in a graduated cylinder and set aside.
 (Note: every time you pour out a solution, touch one glass container to the other to prevent solution spills and to also ensure that all of the solution is removed from one container to the other, if desired.)
- 3. Prepare 20 mL of a 5% by volume solution of adipoyl chloride (e.g. 1 ml of adipoyl chloride in 19 ml of solvent). Use cyclohexane as the solvent and mix gently for 30 seconds. Measure out 10 mL of this solution in a graduated cylinder and set aside. Since this solution has an organic solvent, place a cap over the graduated cylinder.

Add water to sodium hydroxide pellets to make 25 mL of a 20% by weight solution.
 You will need to use the balance to weigh the pellets.

(Caution: the sodium hydroxide will cause skin and eye irritation. Be sure wear gloves while handling and not to touch your eyes or skin after handling. Also, the reaction is exothermic and will produce heat at a temperature over 80 $^{\circ}$ C.)

1. Transfer the 10 mL of 1,6-hexanediamine into a 50 mL beaker and add 10 drops of the sodium hydroxide solution to it.

2. Tilt the beaker with the 1,6-hexanediamine and the sodium hydroxide solution in it at a 45 degree angle and SLOWLY add the 10 mL of the adipoyl chloride solution to it. Two layers will be present since these solutions are not miscible in each other and the polymer will immediately form at the liquid-liquid interface (Figure 1.5).

(Note: HCL acid vapor is harmful when inhaled.)

- 3. Using a copper wire with a hook at the end, slowly go around the inside wall of the beaker to free it of the polymer and in a circular motion, approach the center of the beaker. Gently pull up on the polyamide at the center of the polymer's mass and a continuous rope will form out of the solution. Slowly pull the rope until a few feet of the polymer is out of the solution (if you pull it too fast the rope will break), and then cut the rope off. Lay this rope on a paper towel to dry.
- 4. Quickly stir the remaining solution until a bulk polymer forms inside the beaker.
- 5. Rinse both the bulk polymer and the polymer rope in water three times. Use a paper towel to get dab excess water out of the fiber and then use a piece of filter paper to pull more water out of the fiber. Wrap the polyamide in filter paper and place it aside.

6. Dispose of all organic waste in the "organic waste" container. Dispose of all water-based solution in the container marked "base waste". Clean all glassware with water and put on the rack to dry. Throw away all paper waste. Also clean the balance.

RESULT: The yield of nylon 66 polymer is g.

AIM: Preparation and characterization of polystyrene.

APPARATUS: Dropper, measuring cylinder, beakers, test tubes, glass rod, water bath, etc.

CHEMICALS: Styrene, benzoyl peroxide, sodium hydroxide & anhydrous Calcium chloride.

THEORY: Polystyrene (IUPAC Polyphenylethene) is an aromatic polymer made from the aromatic monomer styrene. It is a thermoplastic polymer, existing in solid state at room temperature, but melts on heating (for moulding), and becomes solid again when cooled. Polymerization is initiated by benzoyl peroxide following free radical addition mechanism.



PROCEDURE:

Solid styrene is taken in a separating funnel and mixed thoroughly with 10% sodium hydroxide solution. Allow the mixture to settle & separate the aqueous alkaline layer. The monomer is washed with aqueous NaOH for two to three times to remove acidic impurities & then it is washed with distilled water to remove alkali. Then at last the monomer is dried with anhydrous CaCl₂. Now the monomer is in its pure form.

Take about 10 gm of pure styrene in a test tube & add 8-9 ml 2% of benzoyl peroxide to it. Close the mouth of the test tube by placing a cotton plug. Then heat the test tube in water bath for 45 mins till solidification occurs. The solid obtained is pure polystyrene.

PROPERTIES:

- 1) Pure solid polystyrene is a colourless, hard plastic with limited flexibility.
- 2) Polystyrene can be transparent or can be made to take on various colors.

- 3) It is having density of 1.05 gm/cc with softening temperature of 90°c
- 4) It produces metallic sound on dropping & is brittle.

5) Resistant towards acids, alkali, oxidizing and reducing agents and moisture.

APPLICATIONS:

- 1) Coploymer with divinylbenzene is used as ion-exchange resin.
- It is economical & used for producing plastic lids, jars, disposable cups, bottles, buttons, combs, license plate frames, plastic cutlery, CD & Jewel cases, ratio & television cabins lenses etc.

3) Foamed polystyrene is used for making disposable cups, packing cases etc.

RESULT: - The yield of polystyrene polymer is g.

EXPERIMENT No. : 06

AIM: Preparation of urea-formaldehyde resin.

APPARATUS: Dropper, measuring cylinder, beakers, test tubes, glass rod, water bath, etc. CHEMICALS: Urea, 40% formaldehyde, concentrated sulphuric acid & distilled water. PRINCIPAL: Urea-formaldehyde, also known as urea-methanal, named so for its common synthesis pathway and overall structure, is a thermosetting resin or polymer, made from urea & formaldehyde. The reaction involves condensation between the nucleophilic nitrogen of urea is formed which further reacts with excess of urea to form a water soluble branched copolymer.

Thermosets: The polymers which on heating change irreversibly into hard rigid and infusible materials are called thermosetting polymers. These polymers are usually prepared by heating relatively low molecular mass, semi fluid polymers, which becomes infusible and form an insoluble hard mass on heating. The hardening on heating is due to the formation of extensive cross-linking between different polymeric chains. This lead to the formation of a 3 Dimnesional network of bonds connecting the polymer chains. Since the 3D network structure is rigid and does not soften on heating, the thermosetting polymers cannot be reprocessed. Some important examples of thermosetting polymers are Phenol-Formaldehyde resin and Melamine-Formaldehyde resins. 

Monomethylol Urea



PREPARATION:

Urea and formaldehyde react with each other in neutral or acidic conditions to give mono and dimethylol urea, which undergo further condensation reaction to give linear, partially cross linked or fully cross-linked polymer.

PROCEDURE:

- 1. Place about 5 of 40% formaldehyde solution in 100 ml beaker.
- 2. Add about 2.5 g of urea with constant stirring till saturated solution is obtained.
- 3. Add a few drops of conc. Sulphuric acid with constant stirring.
- 4. A voluminous white solid mass appears in the beaker.
- 5. Wash the white solid with water and dry it in the folds of filter paper.
- 6. Calculate the yield of the product

RESULT: - The yield of urea formaldehyde resin is g.

- 1. Sulphuric acid should be added drop by drop with constant stirring.
- 2. White solid mass should be removed immediately as it sticks to the walls of the beaker.

3. The reaction mixture should be stirred continuously.

4. The reaction is sometimes vigorous and it is better to be a few feet away from the beaker while adding the H₂SO₄ and until the reaction is complete.

APPLICATIONS:

- 1. Used for making buttons, bottle caps cosmetic container closures etc.
- As a binder of glass fibers, rock wool & plywood. Also used for electrical insulation (switches, boards, desk lamp casing etc.)
- 3. Also used in agriculture as a controlled release source of nitrogen fertilizer.

AIM: Preparation of phenol-formaldehyde resin (Bakelite).

APPARATUS: Conical flask, air condenser, steam bath, thermometer, measuring cylinder, beaker, etc.

CHEMICALS: Phenol, 40 % formaldehyde (formaline) & conc. Sulphuric acid & glacial acetic acid

PRINCIPLE: Phenol is reactive towards formaldehyde at ortho and para sites (sites 2,4 and 6) allowing up to 3 units of formaldehyde to attach to the ring. This forms a hydroxymethyl phenol. The hydroxymethyl group is capable of reacting with either another free ortho or para site, or with another hydroxymethyl group. The first reaction forms a methylene bridge, and the second forms an ether bridge. Phenol formaldehyde resins, are formed by also growth polymerization reaction which may be either acid or base catalysed.

Base catalysed phenol formaldehyde resins are made with formaldehyde to phenol ratio of greater than one (around 1.5). phenol, formaldehyde, water and catalyst are mixed in the desired amount, depending on the resin to be formed, & are then heated. The first part of the reaction, at around 70°C, forms hydroxymethyl phenols. This results in a thick reddish-brown goo, the resin. The negative charge is delocalized over the aromatic ring, activating sites 2,4,&6, which then react with formaldehyde to form hydroxymethyl phenols. Hydroxymethyl phenols will crosslink on heating to around 120°C to form methylene and methyl ether bridge. At this point the resin is starting to cross link, forming highly extended 3-dimensional web

of covalent bonds which is typical of polymerized phenolic resins.

PREPARATION: PFs are prepared by reaction of phenol with formaldehyde in the presence of acidic or basic catalyst. The process may be carried out as follows: A mixture of phenol and formaldehyde are allowed to react in the presence of a catalyst. The process involves formation of methylene bridges in *ortho*, *para* or *both ortho* and *para* positions. This results first in the formation of linear polymer (called *NOVALAC*) and then in to cross-linked polymer called phenol-formaldehyde resin or *bakelite*.



Phenol



Formaldehyde





p-Hydroxy Methyl Phenol





PROCEDURE:

1. Place 5ml of glacial acetic acid and 2.5ml of 40% formaldehyde solution in a 500ml beaker and add 2 grams of phenol.

2. Add few ml of conc. Sulphuric acid into the mixture carefully. Within 5 min. a large mass of plastic is formed.

3. The residue obtained is washed several times with distilled water, and filtered product is

dried and yield is calculated.

RESULT: The weight of the phenol formaldehyde resin is g.

APPLICATION:

1. Resol (methyl phenols) is used as varnish & lacquer for making of laminates.

2. PF is used in electrical circuits & switches, in automobile parts & for making moulded

articles like telephones.

3. Also used as an adhesive for grinding wheels & break lining & in making sand paper.

PRECAUTIONS:

1. The reaction is sometimes vigorous and it is better to be a few feet away from the beaker

while adding the H₂SO₄ and until the reaction is complete.

2. The experiment should be preferably carried out in fume cupboard.

EXPERIMENT No.: 08

AIM: To determine the molecular weight of a given polymer (polystyrene) by using viscometric method.

PRINCIPLE:

Molecular weight of a polymer is nothing but the average molecular weight. This can be determined by measuring the intrinsic viscosity (η_i) of a dilute polymer solution.

This intrinsic viscosity is related to the molecular weight by following relationship

 $\boldsymbol{\eta}_i = \boldsymbol{K} \boldsymbol{M}^{\boldsymbol{a}}$ (Mark-Howink equation)

where η_i = Intrinsic viscosity

K & a =constants for a given polymer-solvent combination at a given temperature.

M = average molecular weight.

PROCEDURE:

Polymer solution of different concentration say 0.5%, 1.0%, 1.5%, 2.0%, 2.5% are prepared from the given polymer stock solution as shown in the table.

DETERMINATION OF MOLECULAR WEIGHT OF POLYMER:

20 ml of the solvent is taken into the viscometer and is sucked through the capillary tube up to the upper mark without any air bubbles. Now note the flow time of the solvent to flow from the upper mark (m_1) to lower mark (m_2) .

Now fill the viscometer with 20 ml of the polymer solution (say I) into the viscometer and determine the flow time of the polymer solution to flow from the upper mark (m_1) to lower mark (m_2) . Using the same procedure, determine the flow time of the various concentrations of polymer solution.

From the flow times, reduced viscosity (η_{sp}/C) can be calculated. Graph is plotted between η_{red} vs. concentration; a straight line is obtained with an intercept called intrinsic viscosity (η_i) .

TABULATION:

Flow time of the solvent $t_0 = \dots$ sec.

| Concentration | Flow time | $\eta_0/\eta = t/t_0 =$ | | |
|---------------|-----------|-------------------------|---------------------------------|------------------------------------|
| C (g/100ml) | 't' sec | ηrel | $\frac{t}{t_o} - 1 = \eta_{sp}$ | $\eta_{red} = \frac{\eta_{sp}}{C}$ |
| 0.5 | | | | |
| 1.0 | | | | |
| 1.5 | | | | |
| 2.0 | | | | |
| 2.5 | | | | |

RESULT: The molecular weight of the given polymer is

EXPERIMENT No.: 09

AIM: To find the eutectic point for two component system by cooling curve method.

APPARATUS: Boiling tube, test tubes, thermometer 360°, Stirrer, beaker, stop watch.

CHEMICALS: Urea, phenol, Colorless liquid (Paraffin/glycerin)

PRINCIPLE:

Eutectic System: A binary system consisting of two substances, which are miscible in all proportions in the liquid phase, but do not react chemically is known as "Eutectic (Easy to melt) system", e.g. A mixture of lead and silver comprises of such a system.

Eutectic Mixture: is a solid solution of two or more substances having the lowest freezing point of all the possible mixture of the components. Example: Alloys of low melting point

Eutectic Point: Two or more solid substances capable of forming solid solutions with each other have the property of lowering each other's freezing point and the minimum freezing point attained, corresponding to the Eutectic mixture, is termed as Eutectic Point (Mean lowest M.P)

Applications of Eutectics

Low melting alloys are used in safety devices (e.g. as plugs in automobiles), fire sprinklers and as fail-safe device in boilers. By suitable choice of metals, very low melting alloys can be obtained. E.g. wood's metal (Alloy containing 50% Bi, 25% Pb, 12.5%, Cd, 12.5% Sn) melts at 65 0 C only.

Congruent melting compound:-

The melting point of phenol is 43 0 C and that of urea is 132 0 C. When they are heated together, they chemically react with each other to form a thermally stable compound which is called congruent melting compound. The congruent melting compound has melting point of 61 0 C and its composition is **Urea: phenol = 1:2**

In thermal analysis curve, congruent melting point is observed as hault i.e.when temperature becomes constant The purpose of this experiment is to obtain data by thermal analysis for constructing a phase diagram which indicates the solid and liquid phase that are present at each temperature and composition. The method consists of cooling of a molten mixture of the components and a graph, known as cooling curve, between temperature and time is drawn. **Cooling curve** is continuous curve as long as there is no phase change during the cooling. When the solid starts separating, the rate of cooling is decreased due to liberation of heat

In order to obtain a phase diagram, mixture of two compounds is prepared. This mixture is heated to a liquid state and then cooling curve is obtained for the mixture.

Thermal analysis:

Thermal analysis is the study of the cooling curves of various compositions of a system during solidification. It is used for finding the shape of the freezing – point curves of any system especially those involving metals. For any mixture of a definite composition, it is possible to find out freezing point and eutectic point from the cooling curves.

Cooling curves



Figure: (a). When a pure substance in the fused state is allowed to cool slowly and the temperature noted at definite intervals. The graph of rate of cooling will be continuous curve. When the freezing point is reached and the solid makes its appearances by the break in the continuity of the cooling curve and the temperature remains constant, until the liquid is fully solidified. Thereafter, the fall in temperature will again become continuous.

Figure: (b). When a mixture of two solids in the fused state is allowed to cool slowly and the temperature noted at definite intervals. The graph of rate of cooling will be continuous curve. When a solid phase begins to form, the rate of cooling curve exhibits a break. However, the temperature does not remain constant as in the previous case of cooling of a pure substance. The temperature decreases continuously until the eutectic point reached. Now the temperature remains constant, till the completion of solidification. Thereafter, the fall of temperature becomes uniform, but the rate of fall is quite different from the previous one.

PROCEDURE

- 1. Prepare a mixture of 2.5g urea and 5g phenol
- 2. Melt the mixture by placing the test tube in the bath of hot liquid paraffin or glycerin
- 3. When the whole mass has melted, remove the tube from the bath, wipe it clean and fit it with cork and thermometer. The tube is then fitted in boiling tube which serves as a air jacket.
- 4. Start the stop watch and note the temperature at different intervals of time (say after every min)
- 5. Allow the temperature to fall still further
- 6. (i.e., allow the cooling to continue) and find out the temperature at which the solid and liquid systems show temperature halts before complete solidification. Lowest temperature halt will be the Eutectic point.

OBSERVATION TABLE

Mixture (Composition: Urea + Phenol = 1:2)

| Time in Min. | Temperature ⁰ C |
|--------------|----------------------------|
| | |
| | |
| | |
| | |

Graph: Draw a cooling curve by plotting time of cooling in minutes along the X-axis and temperature in ⁰C along Y-axis. From the cooling curve find the eutectic temperature of the mixture.

PRECAUTIONS:-

1. Weigh the quantity of urea and phenol accurately to get exact Eutectic mixture

- 2. Read the temperature carefully and accurately
- 3. Handle the phenol carefully.

EXPERIMENT No. : 10

AIM: To determine the distribution coefficient of iodine between CCl₄ and water at room temperature.

PRINCIPLE:

According to Nernst distribution law, when a solute distributes between two immiscible solvents in contact with each other, there exists, for similar molecular species, at a given temperature, a constant ratio of distribution between the two solvents irrespective of the total amount of the solute and irrespective of any other molecular species which may be present. In other words,

$$\frac{C_1}{C_2} = K_{D}$$

where KD is termed as the distribution coefficient, and, the terms C_1 and C_2 refer to the concentrations of similar molecular species in the two liquids at a constant temperature.

Now we are considering a system of distribution of iodine between aqueous and CCl_4 layer in the presence of KI in aqueous layer. Equilibrium between KI and I_2 will be attained in the aqueous layer. The equilibrium attained is

$$KI + I_2 \rightarrow KI3^{-1}$$

The equilibrium constant

$$Kequ = \frac{[KI_3]}{[KI] [I_2]}$$

PROCEDURE:

Serially labeled three bottles are taken and mixtures are made in each bottle as follows: These bottles are stoppered well and shaken in a mechanical shaker for about an hour. After shaking, they are set aside for about 20 minutes in a water trough so that it will attain equilibrium. Exactly 2 ml of the CCl₄ layer from bottle I is pipette out into a conical flask and approximately 20 ml of distilled water is added this biphasic solution is titrated against previously standardized thio solution using starch as indicator. The endpoint is the disappearance of blue color. The experiments are repeated to get concordant values. From bottle I, about 10 ml of the aqueous layer is pipette out and the amount of I_2 is estimated as done with CCl_4 layer. The above procedure is repeated for bottles II and III.

| Bottle No. | Contents |
|------------|---|
| Ι | 20 ml of I_2 in CCl ₄ + 40 ml of water |
| II | 20 ml of I ₂ in CCl ₄ + 40 ml of known KI |
| III | 20 ml of I ₂ in CCl ₄ + 40 ml of unknown KI |

TABULATION:

| Bottle | Volume of CCl ₄ layer | Burette Reading (ml) | | Volume of thio (ml) | Concordant Value (ml) |
|--------|-------------------------------------|-------------------------|---------------|------------------------|--------------------------|
| | (mi) | Initial | Initial Final | | |
| Ι | 2.0 | | | | |
| | 2.0 | | | | |
| Ι | 2.0 | | | | |
| Ι | 2.0 | | | | |
| Ι | 2.0 | | | | |
| Ι | 2.0 | | | | |
| Bottle | Volume of aqueous layer (ml) | Burette Reading (ml) | | Volume of thio (ml) | Concordant Value (ml) |
| | | Initial | Final | | |
| Ι | 10.0 | | | | - |
| | 10.0 | | | | |
| Ι | 10.0 | | | | |
| Ι | 10.0 | | | | |
| I | 10.0 | | | | |
| Ī | 10.0 | | | | |

| Bottle No | Layer | Volume of Thio (ml) | Strength of Iodine | $\frac{C_1}{C_2} = K_D$ |
|-----------|------------------|------------------------|-----------------------|-------------------------|
| Ι | CCl ₄ | | | |
| | Aqueous | | | |
| II | CCl ₄ | | | |
| | Aqueous | | | |
| III | CCl ₄ | | | |
| | Aqueous | | | |
| | | | | |

RESULT: The distribution coefficient of iodine between CCl_4 and water is found to

be

EXPERIMENT NO. : 11

AIM: Identification of functional groups by FT-IR spectroscopy.

Spectroscopy involves measuring the interaction of molecules with electro-magnetic radiation. Essentially the entire electromagnetic spectrum, from γ radiation to radio waves, can be used for studying molecules.

Infrared spectroscopy (IR) uses radiation at wavelengths somewhat longer than visible light. IR radiation has a relatively low energy (a wavelength of 1 μ m corresponds to 120 kJ/mol, which is considerably less than covalent bond strength). As with all absorption phenomena, the absorption of IR radiation only occurs when the energy of the radiation corresponds to that required for a specific transition in the molecule. Molecules absorbing IR radiation undergo transitions in vibrational modes. Because each functional group in an organic molecule has characteristic vibrational transitions, IR spectroscopy is a very useful technique for identifying functional groups in organic molecules. IR absorption requires a **change in the dipole of the molecule** as a result of the vibrational mode, with larger dipole changes resulting in more intense bands.

The intensity of a peak in an IR spectrum is proportional to:

- 1. The number of molecules present in IR beam (which means that both higher concentrations and thicker films result in more intense bands).
- 2. The amount of a particular bond-type in the molecule (for example, the C–H stretch is usually intense, because most organic compounds have many C–H bonds).
- 3. The polarity of the bond (more polar bonds result in more intense IR bands).

Wavenumber (cm⁻¹) is a frequency measurement, corresponding to the number of wave peaks in one centimeter. Some older sources use "microns", which are wavelength units.¹⁰ The IR spectrum most commonly used extends from ~4000 cm⁻¹ to ~500 cm⁻¹ (~2.5 to ~16 μ m).

IR spectra are generally reported using wavenumbers on the *x*-axis, with higher numbers on the left, and using % Transmittance on the *y*-axis. In parts of a spectrum where a sample lacks IR signals, the % Transmittance will be large (because the majority of the IR radiation reaches the detector). Peaks extend downwards from the baseline.

Performing IR Measurements

In performing IR measurements, it is necessary to place the sample into the spectrometer. Unfortunately, most liquid and solid compounds absorb IR radiation very strongly, and a test tube full of a sample would be opaque in the IR. The standard method involves the use of thin films of liquid sample held between "salt plates". **Salt plates** are crystals of sodium chloride; sodium chloride crystals do not absorb IR radiation at wavenumbers higher than ~600 cm⁻¹. *Salt plates are fragile!* Handle the plates with care, and clean the plates with acetone (*do not use water to clean the plates!*). The salt plates should be stored in a dessicator when not in use to prevent damage due to water in the air.

Performing an IR measurement on a liquid sample is simple (the technical term is a "neat spectrum"). The instructions below are specific for the *Easy Omnic* software that runs the FTIR instruments in the Moench F108 Organic Chemistry Lab, although many of the steps will be similar for any IR spectrometer.

- 1. Check to be sure that the sample chamber is empty.
- 2. If it is not already running, run the Omnic or EZ Omnic software.
- 3. Click the Col Bkg (Collect Background) button. When the background spectrum is complete, click No in the Add to New Window dialog.
- 4. Remove a pair of salt plates from the dessicator.
- 5. Place one or two drops of your liquid sample on one salt plate, and immediately place the second plate on top. This should create a thin film of liquid between the plates. (If the liquid runs off the plate, it may form a "thick" film on the opposite

6. side of the plate, which may make it difficult to obtain a good spectrum).

- 7. Place the salt plate sandwich in the sample holder, and place the holder in the sample chamber. (*The clip on the sample holder should be on top* when the holder is in the instrument; otherwise, the hole in the holder will not align with the optical source and detector.
- 8. Click the Col Smp (Collect Sample Spectrum) button.
- After the spectrum is complete, click Yes to add to new window, and then click Find Pks (Find Peaks), and then Replace.
- 10. Print the spectrum.
- 11. Clean the plates with acetone, and allow the acetone to evaporate.
- 12. Return the plates to the dessicator.

The thickness of the film is important. Pitted plates give poor spectra, because the relatively thick film will result in some of the peaks being off-scale. Clear samples give good, flat baselines. Cloudy, turbid samples tend to have baseline problems.

Any compound that is present will contribute to the spectrum. Try to *stand back from the instrument while collecting data, because water vapor and carbon dioxide in your exhalations will appear in your spectra*. In addition, make sure that any acetone used to clean the plates has evaporated before you use them, and that your sample does not contain water contamination.

IR on Solids

You cannot place a solid sample on a salt plate and obtain a usable spectrum. Instead, you must either make your own plate (using anhydrous potassium bromide; ask your instructor to show you how to do this if necessary) or obtain the spectrum of a solution of your sample. For the compounds you are likely to use, a chloroform (CHCl₃) solution is useful because chloroform has only four significant IR peaks, and because most (although not all) organic compounds dissolve readily in chloroform. To create the sample, either add CHCl₃

to a few crystals on a salt plate, or (preferably) dissolve the sample in CHCl₃ in a test tube. Use a minimum volume of chloroform, because *a* more concentrated sample usually yields a better spectrum. Once you have a chloroform solution, obtain the spectrum normally. (Note: some alacrity in placing the sample on the salt plates and collecting the spectrum is desirable, because as the chloroform evaporates, the solute may precipitate from the solution and cause serious baseline problems.)



Figure: The IR spectrum for chloroform.

A full explanation of IR spectral interpretation is beyond the scope of this lab manual. However, an abbreviated IR correlation table is shown on the next page.

Chloroform is an anesthetic and a liver toxin; avoid breathing the vapors. In addition, chloroform is a halogenated compound, so be sure to dispose of excess chloroform in the appropriate waste.

Table - IR Absorption Bands of Functional Groups in Organic Compounds

| Functional | Wave- | Comments |
|--------------------|---------------------|--|
| Group | Number | Comments |
| F | (cm ⁻¹) | |
| | | |
| | 2850-3000 | C–H stretch for sp ³ carbon (Strong) |
| | 1450-1470 | C–H bend (Strong) |
| Alkane | 1370-1380 | C-H methyl umbrella bend (Medium) |
| | 800-1200 | C–C stretch (many medium bands) |
| | 720-725 | C–H bend (Medium) |
| | 3050-3140 | C–H stretch for sp ² carbon (Medium) |
| Alkene | 1620-1680 | C=C stretch (Medium) |
| | 900-1000 | C–H bend (Strong) |
| Terminal alkyne | 3300-3320 | C–H stretch for sp carbon (Medium) |
| (internal alkynes | 2100-2140 | C=C stretch (medium) |
| lack unique bands) | 600-700 | C–H bend (Strong) |
| 1 | 3200-3600 | H–bonded O–H stretch (Broad strong |
| | | band) |
| | 3600-3700 | Free O–H stretch (sharp medium band, present |
| Alcohol | 2000 2700 | only in dilute samples) |
| | | 5 |
| | 1000-1200 | C–O stretch (strong, higher v for more |
| | 1000 1200 | substituents on carbon) |
| Ether | 1050-1150 | C–O stretch |
| Primary amine | 3200-3400 | N-H stretch (two medium bands) |
| Secondary amine | 3200-3400 | N_H stretch (single medium band) |
| Nitrilo | 2200-3400 | N=C stretch (modium) |
| Niulle | 1700 1720 | N=C stretch (fitnenge) (leven a for conjugated) |
| Aldahyida | 1/00-1/20 | C=O stretch (Strong) (lower v for conjugated) |
| Aldellyde | 2700 2850 | Aldehude C. Histrateh, two modium hands |
| Votono | 1700-2830 | Aldenyde C-II stretch, two incutani bands |
| Ketone | 1700-1720 | C=O stretch (Strong) (lower v for conjugated) |
| 0 1 1 1 | 1700-1720 | C=O stretch (Strong) (lower v for conjugated) |
| Carboxylic acid | 3000 | O-H stretch (very broad band, usually with a a^{2} C. It stretch is the widdle) |
| | | spike of sp ⁵ C–H stretch in the middle) |
| | 1730-1750 | C=O stretch (Strong) (lower v for conjugated) |
| Ester | 1000 1000 | |
| | 1000-1300 | C–O stretch |
| A 11 | 1650-1690 | C=O stretch (Strong) (lower v for conjugated) |
| Amide | 2200 2400 | |
| | 3200-3400 | N–H stretch (two bands for primary, one for |
| | | secondary, none for tertiary amide) |
| | 3000-3150 | C–H stretch (variable) |
| | 1750-1950 | C–H bending overtone region (usually several |
| Aromatic | | |
| | | weak bands of similar strength) |
| | 600-900 | C–H bend (strong) |
| | 1550 and | "Walrus teeth" N–O stretch bands |
| Nitro | | |
| | 1400 | |
| | | |

EXPERIMENT NO. : 12

AIM: To determine $\lambda_{(max)}$ (wave length of maximum absorption) of solution of KMnO4 using a Spectrophotometer.

APPARATUS: UV – visible spectrophotometer, beaker.

CHEMICALS: Distilled water, standard solution of KMnO4, tissue paper.

PRINCIPLE:

When an electromagnetic radiation is passed through a sample, certain characteristic wavelengths are absorbed by the sample. As a result the intensity of the transmitted light is decreased. The measurement of the decrease in intensity of radiation is the basis of spectrophotometer. Thus the spectrophotometer compares the intensity of the transmitted light with that of incident light.

The absorption of light by a substance is governed by certain laws.

According to the Beer Lambert's law the intensity of the incident light is proportional to the length of thickness of the absorbing medium and the concentration of the solution,

$$\log I_0/I = A = \varepsilon cL$$

 I_0 = Intensity of incident light

I = Intensity of transmitted light

A = Absorbance

- L = Thickness of the medium
- $c = Concentration in moll^{-1}$
- ε = Molar absorption coefficient

(OD). The absorbance (OD) of a solution in a container of fixed path length is directly proportional to the concentration of a solution. i .e

$$A = \mathcal{E}cL$$

A plot between absorbance and concentration is expected to be linear. Such a straight line plot, passing through the origin, shows that Beer – Lambert's law is obeyed. This plot, known as calibration curve can be employed in finding the concentration of a given solution.

SPECTROPHOTOMETER:

A spectrometer is a device which detects the percentage transmittance of light radiation when light of certain intensity and frequency range is passed through the sample. Thus the instrument compares the intensity of the transmitted light with that of the incident light. There are many spectrophotometers available for the visible range extending from 3800-7800 A^0

Setting of the Spectrophotometer

- Spectrophotometer should initially read zero on transmittance scale (T). If it does not read zero, set it mechanically with adjusting knob.
- 2) Connect the instrument to the mains and put on the power switch.
- 3) Adjust the wavelength knob to the required wavelength region on scale.
- Choose the position of wavelength switch correspondingly either to 340 400 nm or 400-960nm.

5) Adjust the meter needle on zero transmittance scale and 100 on O.D scale.

Working of the Spectrophotometer

- Open the lid of the cell compartment and insert a cuvette containing the blank solvent (distilled water). Close the lid.
- 2) Adjust the needle to 100% transmittance or zero optical density.
- 3) Remove the cuvette and close the lid tightly again. Empty the cuvette and rinse it with the standard solution of KMnO4 (0.01M). Fill it with standard solution.
- Now place the cuvette containing the standard solution in the cell compartment. Note the O.D and transmittance.

- 5) Now change the wavelength by 20nm and note absorbance (OD) and transmittance for each wavelength.
- Plot a graph between wavelength measurement on the x-axis and absorbance (OD) on the y-axis.

OBSERVATION:

Determination of λ_{max}

TABLE:

| Wavelength (nm) | Absorbance(OD) |
|-----------------|----------------|
| | |
| | |
| | |
| | |

- i) A curve is plotted between wavelength and absorbance (OD).
- ii) A curve is plotted between O.D and concentration and a straight line is obtained as shown by equation.

iii) From the graph of O.D versus concentration, the concentration of the unknown solution can be found out. For example, in the fig x is the O.D of unknown solution then its concentration will be 1.0%.

RESULT:

 λ_{max} for KMnO4 solution =.....nm

PRECAUTIONS:

- i) Always use dilute solutions for getting calibration curve.
- ii) Cuvette should be cleaned properly and must be wiped with tissue paper.
- iii) Do not leave any finger marks on the cuvette.

EXPERIMENT NO. : 13

AIM: Determination of concentration of an unknown sample by UV spectroscopy.

PRINCIPLE:

According to Beer-Lamberts law, the optical density of absorbance of a solution of concentration 'c' mol dm⁻³ placed in a cell of 'b' cm width is given by

$A = \varepsilon bc$

where ε is called the molar absorption coefficient or molar extinction coefficient. The absorbance **A** is defined as

$$\mathbf{A} = \log\left(\mathbf{I}_0/\mathbf{I}\right)$$

where I_0 and I represents the intensities of incident and transmitted radiations, since the optical density is linearly proportional to the concentration of the solution, a linear plot is expected for absorbance Vs concentration.

Copper (II) forms a colored complex with $K_4[Fe(CN)_6]$ by the reaction,

$$Cu^{2+} + K_4[Fe(CN)_6] \longrightarrow Cu_2[Fe(CN)_6] + 4K^+$$

This complex absorbs bluish green light of wavelength maximum 480 nm and therefore exhibits its complementary color. The absorbance of this complex can be measured by using a spectrocolorimeter fixing max at 480 nm. A calibration line is plotted by measuring the optical density of the standard solution of various concentrations. The concentration of the unknown is determined by matching its optical density in the calibration curve.

PROCEDURE:

0.2 g of CuSO₄ is weighed accurately in a chemical balance transferred into a 100 ml SMF and made up to the mark using distilled water. A drop of concentrated H₂SO₄ is

added to prevent precipitation of $Cu(OH)_2$. This solution is approximately diluted to 10 times in a 100 ml SMF. 1 ml of this solution is taken in a test tube. 5 ml of 10% NH_4NO_3 and 1 ml of 4% $K_4[Fe(CN)_6]$ are added and made up to 15 ml. The optical density of this solution is measured using a spectrocolorimeter after fixing maximum wavelength at 480 nm. Similar measurements are made with 2, 3, 4, 5, 6 and 7 ml of standard $CuSO_4$ solution and the calibration line is obtained. From the calibration line, the amount of copper (II) present in the unknown is determined using its optical density.

TABULATION:

| S.No | Volume of CuSO4.5H2O solution (ml) | Optical density | Conc. in gm |
|------|---------------------------------------|-----------------|-------------|
| 1 | 1 | | |
| 2 | 2 | | |
| 3 | 3 | | |
| 4 | 4 | | |
| 5 | 5 | | |
| 6 | 6 | | |
| 7 | 7 | | |

RESULT-The amount of copper (II) present in unknown sample is gm.

EXPERIMENT NO. : 14

AIM: Determination of coefficient of viscosity of the given liquids by Ostwald's viscometer method.

APPARATUS: Ostwald viscometer, rubber tube with screw pinch cock, relative density (R.D) bottle (known as pyknometer also), stand, beaker, unknown liquid, distilled water. **Theory:** The force of friction which one part of the liquid offers to another part of the liquid is called viscosity. For measuring the viscosity coefficient, Ostwald viscometer method is used which is based on Poiseuille's law. According to this law, the rate of flow of liquid through a capillary tube having viscosity coefficient, η , can be expressed as

$$\eta = \frac{\pi \cdot r^4 t P}{8 \text{vl}}$$

where, v= vol. of liquid (in ml)

t= flow time (in sec.) through capillary r= radius of the capillary (in cm) l= length of the capillary (in cm) P= hydrostatic pressure (in dyne/sq.cm) η= viscosity coefficient (in poise).

Since, the hydrostatic pressure (the driving force) of the liquid is given by P = dg h (where h is the height of the column and d is the density of the liquid);

 $\eta \propto P t$; or, $\eta \propto d g h t$

If, η_1 and η_2 are the viscosity coefficients of the liquids under study, d_1 , d_2 , are their densities and t_1 and t_2 are their times of flow of *equal volume* of liquids through the same capillary respectively, then

 $\eta_1 \propto d_1 \; g \; h \; t_1 \; and \; \eta_2 \propto d_2 \; g \; h \; t_2$

 $\frac{\eta_1}{\eta_2} = \frac{d_1 t_1}{d_2 t_2}$

Hence,

Here, usually the viscosity of given liquid is measured with respect to water whose viscosity is known very accurately at different temperatures. The SI physical unit of viscosity is the pascal-second (**Pa** \cdot s), (i.e., kg \cdot m⁻¹ \cdot s⁻¹). This means: if a fluid with a

So, weight of water $(w_w) = (w_2 - w_1) = \dots g$.

Weight of R.D.bottle with water $(w_2) = \dots g$.

Weight of R.D. bottle with liquid $(w_3) = \dots g$.

Weight of empty R.D.bottle $(w_1) = \dots g$.

 $=\frac{(Mass)_{liq}}{(Mass)_{water}}$ (since, volume is same for all) $\left(\frac{Mass}{Vol}\right)_{liq}$ Relative density =

Observations:

Procedure:

1. Laboratory temperature=.... °C

2. Density measurement:

2. Wash the R.D.bottle with distilled water and dry.

3. Take the weight of the empty & filled (with distilled water) R.D. bottle (with stopper). Then, weigh the R.D. bottle filled with unknown given liquid. Use the data for measuring the densities.

4. Clean and rinse the viscometer properly with distilled water. Fix the viscometer vertically on the stand and filled with specific amount (say 20ml) of mixture (every time take the same volume).

5. Time of flows were recorded for each solutions (water and the given liquid).

1. Note the laboratory temperature.

6. Take 3 to 4 readings.

Pa·s]. Water at 20 °C has a viscosity of 1.0020 cP.

viscosity of one **Pa** s is placed between two plates, and one plate is pushed sideways with a shear stress of one pascal, it moves a distance equal to the thickness of the layer between the plates in one second. The cgs unit for the same is the **poise** (P), (named after J. L. Marie Poiseuille). It is more commonly expressed, as **centipoise** (**cP**). [1 cP = 0.001]

3. Combined table for the measurement of flow times and relative densities:

| Sl no. | Flow times (sec) | | | |
|--------|------------------|----------------|----|------|
| | t ₁ | t ₂ | t3 | mean |
| 1 | | | | |
| 2 | | | | |
| 3 | | | | |
| 4 | | | | |

Calculations:

1. Determination of the density of the liquid (d_l):

 $\frac{\text{Density of liquid } (d_l)}{\text{Density of water } (d_w)} = \frac{\text{Weight of liquid } (w_l)}{\text{Weight of water } (w_w)}$

Density of liquid $(d_l) = \frac{w_l}{w_w} d_w$ (Take density of water =1.0g/ml at 25 °C)

2. Determination of the viscosity of the liquid (η_l)

Viscosity of the liquid, $\eta_l = \frac{t_l d_l}{t_w d_w} \eta_w$

Result: The viscosity of the given liquid with respect to water at laboratory temperature was found to becP.

EXPERIMENT NO. : 15

AIM: Investigation of rusting of iron in different conditions.

APPARATUS: Beakers, metal nails, pH – meter, sand paper, weighing balance, etc.

CHEMICALS: HCl, H₂SO₄, H₃PO₄, HNO₃, CH₃COOH, NaOH and KOH

PRINCIPLE: Corrosion of metal can be considered as extractive metallurgy. Rusting is a term reserved for steel and iron corrosion, although many other metals form their oxides when corrosion occurs. Practically all environments are corrosive to some degree. Air, moisture, fresh distilled, salt and mine waters, rural or urban and industrial atmosphere, steam and other gases such as NH₃, Cl₂, H₂S, SO₂, fuel gases, inorganic acids, organic acids, solvents, petroleum oils cause corrosion environment. Inorganic materials are more corrosive than the organics. High temperature and pressure involve more severe corrosion conditions.

If a piece of metal is immersed in a polar solvent like water, some of the metal ions leave the metal surface and go into the solution i.e., dissolution of metal starts. The metal continues to dissolve, more and more electrons are left back and a net negative charge is built up in the metal. Thus metal acquires a negative charge. The potential developed can be measured under standard conditions using standard hydrogen electrode as a reference electrode. It is called standard oxidation potential of metal. The metals having positive values are noble, they do not dissolve readily (metals like gold, platinum, silver, etc.). The metals having negative values of electrode potential are called active metals, which go into solution readily (like iron, zinc, magnesium, etc.).

PROCEDURE: Iron nails were used and were dipped in the different solutions. Take seven 50 ml beakers, label them and take about 30 ml of solutions in the beaker. Dip the

previously weighed iron nails in the beaker such that $4/5^{\text{th}}$ portion is immersed in the solution. Note down the time.

After one week take out the metallic plates from the beaker, dry them in air and then weigh. Note down the respective weights of the plates.

OBSERVATION TABLE:

| S. No. | Medium | Weight loss of iron (mg) |
|--------|-----------------------------------|--------------------------|
| 1 | 4N HCl | |
| 2 | 4N H ₂ SO ₄ | |
| 3 | 4N HNO ₃ | |
| 4 | 4N H ₃ PO ₄ | |
| 5 | CH ₃ COOH | |
| 6 | NaOH | |
| 7 | КОН | |

CALCULATION:

Loss in weight (in 1 week) = before weight – after weight = X gm

RESULT: The rusting of iron nail increases with increasing concentration of acid and with

increase in time.

EXPERIMENT NO.: 16

AIM: Investigation of the effect of metal coupling on rusting of iron.

REQUIREMENT : Two petri dishes, four test – tube , four iron nails, beakers, sand paper, wire gauge, gelatin, copper, zinc & magnesium strips, potassium ferricyanide solution, phenolphthalein.

PRINCIPLE: Rusting may be explained by an electrochemical mechanism. In the presence of moist air containing dissolved oxygen or carbon dioxide, the commercial iron behave as if composed of small electrical cells. At anode of cell, iron passes into solution as ferrous ions. The electron moves towards the cathode and form hydroxyl ions. Under the influence of dissolved oxygen the ferrous ions and hydroxyl ions interact to form rust, i.e., hydrated ferric oxide.

Metal coupling affects the rusting of iron. If the nail is coupled with a more electro-positive metal like zinc, magnesium or aluminum rusting is prevented but if on the other hand, it is coupled with less electro -positive metals like copper, the rusting is enhanced.

PROCEDURE: At first we have to clean the surface of iron nails with the help of sand paper. After that we have to wind zinc strip around one nail, a clean copper wire around the second & clean magnesium strip around the third nail. Then to put all these three and a fourth nail in Petri dishes so that they are not in contact with each other. Then to fill the Petri dishes with hot agar agar solution in such a way that only lower half of the nails are covered with the liquids. Cover Petri dishes for one day or so. The liquids set to a gel on cooling. Two types of patches are observed around the rusted nail, one is blue and the other

pink. Blue patch is due to the formation of potassium ferro-ferricyanide where pink patch is due to the formation of hydroxyl ions which turns colorless phenolphthalein to pink.

OBSERVATION:

| S. No. | Metal Pair | Color of the patch | Nails rusts or not |
|--------|-----------------|--------------------|--------------------|
| 1 | Iron- Zinc | | |
| 2 | Iron -Magnesium | | |
| 3 | Iron- Copper | | |
| 4 | Iron – Nail | | |

RESULT: Coupling of iron with more electropositive metals such as zinc and magnesium resists corrosion and rusting of iron. Coupling of Iron with less electropositive metals such as copper increases rusting